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PREPARATION AND CHARACTERIZATION OF CO(II)/ZRO2 SOLID  
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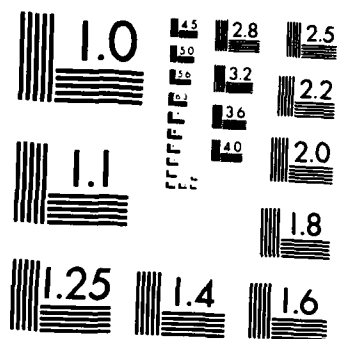
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TECHNICAL REPORT No. 9

Preparation and Characterization of Co(II)/ZrO<sub>2</sub> Solid Solution

by

Ping Wu, Robert Kershaw, Kirby Dwight and Aaron Wold

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## PREPARATION AND CHARACTERIZATION OF Co(II)/ZrO<sub>2</sub> SOLID SOLUTION

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### ABSTRACT

Samples of cubic ZrO<sub>2</sub> containing up to almost 10 atomic percent cobalt were prepared by codecomposition of the nitrate. Magnetic susceptibility measurements confirmed the limit of solubility and the presence of the cobalt as Co(II). The reduction of the cobalt inserted in cubic zirconia took place at a considerably higher temperature than bulk cobalt oxide.

MATERIALS INDEX: Cobalt stabilized cubic ZrO<sub>2</sub>.

### Introduction

Cubic ZrO<sub>2</sub> samples containing some transition metal oxides including rhodium oxide (1), iron oxide (2), chromium oxide (3) and nickel oxide (4) were studied previously in this laboratory. Their structures, magnetic properties and the stabilities towards reduction have been studied. There is little published data concerning the properties of the members in the cobalt oxide-zirconium oxide system. Structural studies of the supported Co(II)/ZrO<sub>2</sub> catalysts were carried out by Bettman and Yao (5, 6). Their samples were prepared by incipient wetness methods and the crystal structure of the products indicated the presence of monoclinic ZrO<sub>2</sub>. Previous work has indicated that the presence of monoclinic ZrO<sub>2</sub> is characteristic of an absence of reaction between the ZrO<sub>2</sub> support and the catalyst.

It is the purpose of this work to investigate the formation of solid solution between ZrO<sub>2</sub> and cobalt oxide and to study the magnetic properties of such a system. In addition, the stability towards reduction of cubic ZrO<sub>2</sub> samples containing various concentrations of reacted cobalt oxide will be determined.

### Experimental

Bulk cobalt oxide samples were prepared by decomposition of Co(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O. The nitrate was dissolved in water and dried at 150°C for 12 hours. The samples were then ground and decomposed at temperatures ranging from 500°C to 800°C. X-ray analysis of the final products indicated the formation of Co<sub>3</sub>O<sub>4</sub>.

Zirconium oxide samples, containing various percentages of cobalt, were prepared by dissolving the desired quantity of analyzed cobalt nitrate  $\text{Co}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  with appropriate amounts of  $\text{ZrO}(\text{NO}_3)_2$ . Two ml of water were added for each millimole of total nitrates. The solution was then dried at  $150^\circ\text{C}$  for 12 hours and the product was ground and heated at  $500^\circ\text{C}$  for 24 hours. Some samples were then subsequently heated at elevated temperatures between  $500$ - $800^\circ\text{C}$ . In order to ascertain the temperatures for complete decomposition of the nitrates, preliminary decomposition experiments were carried out in a Cahn system 113 thermal balance.

X-ray powder diffraction patterns of the samples were obtained using a Philips diffractometer and monochromated high intensity  $\text{CuK}\alpha_1$  radiation ( $\lambda = 1.5405\text{\AA}$ ). For qualitative identification of the phases present, the diffraction patterns were taken in the range  $12^\circ < 2\theta < 80^\circ$  with a scan rate of  $1^\circ 2\theta/\text{min}$  and a chart speed of 30 inches/hr. The scan rate used to obtain x-ray patterns for calculation of cell parameters was  $0.25 2\theta/\text{min}$  with a chart speed of 30 in/hr. Cell parameters were obtained from a least squares refinement of the data with the aid of a computer program which corrected for the systematic experimental errors.

Magnetic susceptibilities were measured using a Faraday balance at a field strength of 10.4 kOe. Honda-Owens (field dependency) plots were also made and all magnetic susceptibility data were corrected for core diamagnetism. Magnetic susceptibility measurements were made from liquid nitrogen temperature to 315K.

Temperature programmed reductions were carried out in a thermal balance equipped with a magnet (7). The weight of the sample was determined using a Cahn electrobalance (model RG) alternately in a magnetic field gradient and without the magnetic field gradient. The temperature was measured by a type S thermocouple which was positioned just below the sample. This technique combines magnetic measurements with thermogravimetric analysis and is very sensitive to the appearance and growth of a magnetic phase during the reaction (7). An 85%Ar/15% $\text{H}_2$  mixture was predried by  $\text{P}_2\text{O}_5$  and passed at a rate of 60 ml/min into the TGA balance. The samples were heated at  $50^\circ\text{C}$  per hour.

### Results and Discussion

Bulk samples of cobalt oxide were prepared by the decomposition of  $\text{Co}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  at temperatures ranging from  $500^\circ\text{C}$  to  $800^\circ\text{C}$ . X-ray diffraction analysis of the products indicated the presence of a single phase, namely  $\text{Co}_3\text{O}_4$ . In order to analyze any deviation from stoichiometry, temperature programmed reduction was carried out by thermogravimetric analysis. The weight change in the process of T.P.R occurs between  $230^\circ$  and  $320^\circ\text{C}$ . The total observed weight change is 26.6%, whereas the calculated weight change of the reduction of  $\text{Co}_3\text{O}_4$  to cobalt metal is 26.5%. These results indicate that the composition of bulk cobalt oxide prepared under these conditions is  $\text{Co}_3\text{O}_4$ .

Samples of the cobalt-zirconium oxide system were prepared by the codecomposition of  $\text{Co}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{ZrO}(\text{NO}_3)_2$ . X-ray analyses of products containing varying compositions are given in Table 1. All of the products reported in Table 1 were prepared at  $500^\circ\text{C}$ .

Decomposition of pure zirconyl nitrate resulted in the formation of tetragonal  $\text{ZrO}_2$  containing a small quantity of monoclinic  $\text{ZrO}_2$ . For the samples prepared by codecomposition of the nitrates, x-ray analysis indicated that  $\text{ZrO}_2$  crystallized with a cubic structure even when only 5 atomic percent of cobalt was introduced into the  $\text{ZrO}_2$ . This is consistent with previous reports (8) that the stabilization of cubic  $\text{ZrO}_2$  requires the presence of a solid solution with cobalt oxide. Products containing up to 10 atomic percent of cobalt showed no evidence of bulk cobalt oxide in the x-ray diffraction patterns. However, it can be seen from Table 1 that there is a decrease in the cell parameter of the cubic  $\text{ZrO}_2$  phase which is consistent with an increase in the cobalt content of

TABLE 1  
IDENTIFICATION OF PHASES FORMED IN THE  $\text{Co(II)}/\text{ZrO}_2$  SYSTEM

Composition (at% Co)	Phase(s)	X-ray Parameters	
		a	c/a
$\text{ZrO}_2$ = 0	Tetragonal $\text{ZrO}_2$ plus small amount amount monoclinic $\text{ZrO}_2$	5.081(3)	1.02
$\text{Co}/(\text{Co}+\text{Zr})$ = 5	Cubic $\text{ZrO}_2$	5.086(3)	
$\text{Co}/(\text{Co}+\text{Zr})$ = 10	Cubic $\text{ZrO}_2$	5.072(3)	
$\text{Co}/(\text{Co}+\text{Zr})$ = 15	Cubic $\text{ZrO}_2$ + $\text{Co}_3\text{O}_4$		

this phase. When attempts were made to prepare cubic zirconium oxide containing 15 atomic percent cobalt, bulk  $\text{Co}_3\text{O}_4$  was evident in the x-ray diffraction patterns. The limit of solubility of cobalt in  $\text{ZrO}_2$  is therefore below 15 percent.

A sample of  $\text{ZrO}_2$  containing 5 atomic percent cobalt was heated to  $600^\circ$ ,  $700^\circ$  and  $800^\circ\text{C}$ . The cubic zirconium oxide remains stable at  $600^\circ\text{C}$  but at  $700^\circ\text{C}$  lines of  $\text{Co}_3\text{O}_4$  and tetragonal  $\text{ZrO}_2$  appear in the diffraction patterns of the products. Finally the monoclinic structure is obtained as the temperature is raised from  $700^\circ$  to  $800^\circ\text{C}$ .

The reciprocal magnetic susceptibility versus temperature data for bulk  $\text{Co}_3\text{O}_4$  (i.e.  $\text{Co(II)[Co(III)}_2\text{O}_4]$ ) is given in Fig. 1. The sample measured has a moment of 4.8 BM per  $\text{Co(II)}$ , since low-spin  $\text{Co(III)}$  is diamagnetic. This is consistent with the value reported in the literature (9). Strong spin-orbit coupling is a characteristic of  $\text{Co(II)}$ , which usually does not show a spin-only moment.

Magnetic measurements were also made on zirconium oxide samples containing 5 and 10 atomic percent cobalt. These measurements were made as functions of both

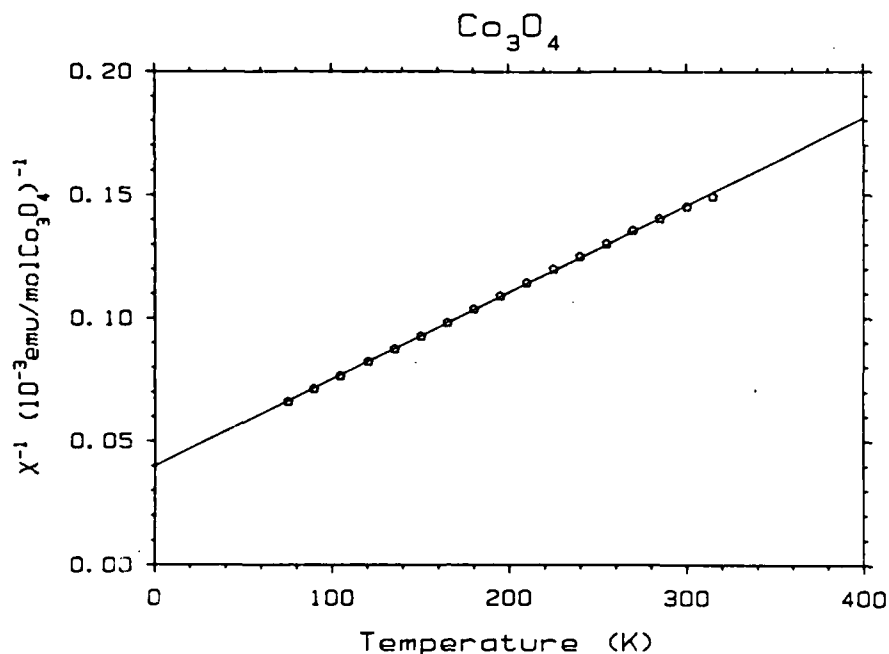


Fig. 1. Reciprocal magnetic susceptibility versus temperature data for bulk  $\text{Co}_3\text{O}_4$  heated at  $500^\circ\text{C}$  for 24 hours.

field and temperature. All samples showed paramagnetic behavior and have no field dependency at either room temperature or liquid nitrogen temperature. The results of the magnetic measurements are plotted in Fig. 2 as reciprocal susceptibility versus temperature. The measured paramagnetic moment for the sample containing 5 atomic percent cobalt is 4.8 BM and hence all of the cobalt present is  $\text{Co(II)}$ . The measured value for the paramagnetic moment per cobalt ion of the sample containing 10 atomic percent cobalt is 4.4 BM which may indicate the coexistence of a small quantity of bulk  $\text{Co}_3\text{O}_4$ . Additional evidence to substantiate these results follow from the TPR studies.

The temperature programmed reduction studies were carried out in a 85% $\text{Ar}/15\%\text{H}_2$  atmosphere using a thermomagnetic balance. The onset of reduction was detected by the difference in sample weight with and without a magnetic field gradient and arises from the ferromagnetism of cobalt. In the case of bulk  $\text{Co}_3\text{O}_4$ , cobalt metal begins to form at  $270^\circ\text{C}$  (Fig. 3), and the sample with 5 atomic percent cobalt in  $\text{ZrO}_2$  begins to reduce at  $540^\circ\text{C}$ . The stability of cobalt oxide towards reduction is therefore greatly increased by forming solid solution with zirconium oxide.

TPR of the sample containing 10 atomic percent cobalt indicated that there were two kinds of cobalt present in the sample (Fig. 3). Most of the cobalt was reduced above  $500^\circ\text{C}$  whereas a small amount of the cobalt began to reduce at  $280^\circ\text{C}$ . Therefore most of the cobalt has been inserted into the  $\text{ZrO}_2$  but a small amount remains as bulk cobalt oxide. This is consistent with the observed decrease of the paramagnetic moment for the 10 atomic percent sample compared to



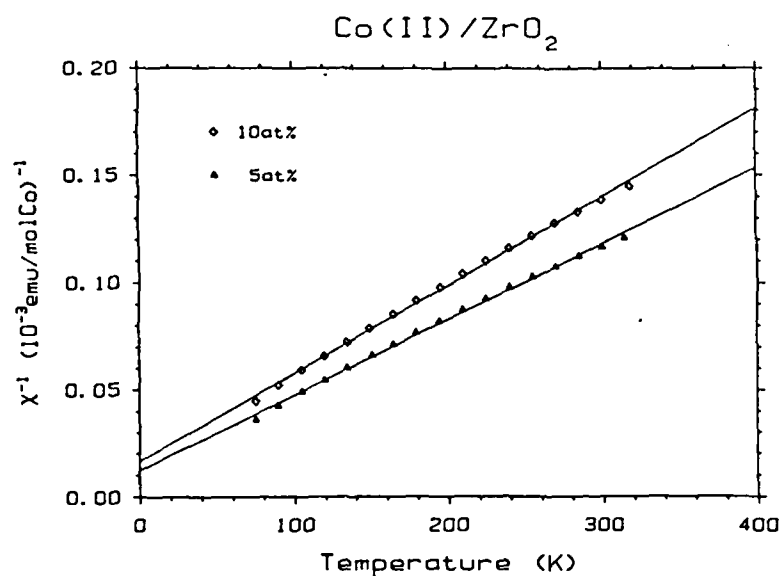


Fig. 2. Reciprocal magnetic susceptibility vs temperature for 5 and 10 at% Co(II)/ZrO<sub>2</sub> heated at 500°C for 24 hours.

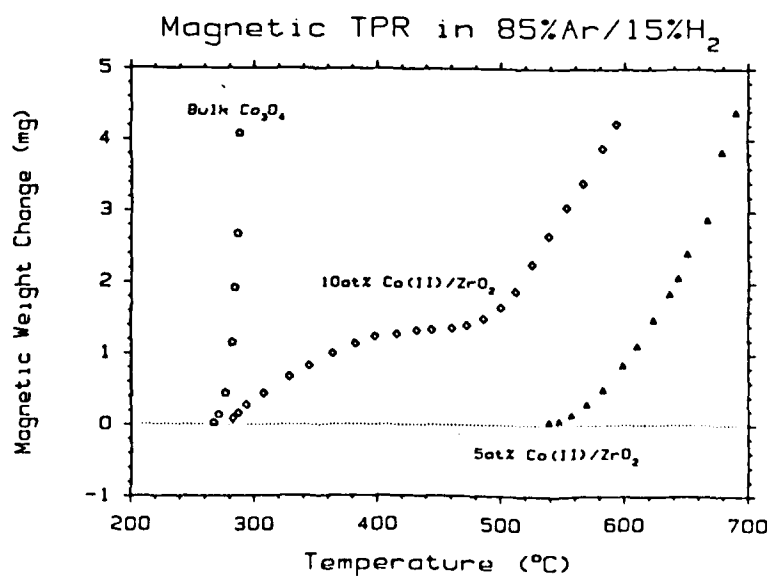


Fig. 3. Variation with temperature of the weight change caused by the application of a magnetic field gradient during the temperature programmed reduction of bulk Co<sub>3</sub>O<sub>4</sub>, 5 and 10 at% Co(II)/ZrO<sub>2</sub> in 85% Ar/15%H<sub>2</sub>.

that obtained for the 5 atomic percent sample. The maximum solubility of cobalt oxide in zirconium oxide is therefore determined to be slightly lower than 10 atomic percent. It appears that codecomposition of the nitrates results in phases which contain cobalt (up to almost 10 atomic percent cobalt) as an integral part of the phase. However, it is only at 15 atomic percent that the presence of bulk cobalt oxide appears in the x-ray diffraction pattern. This is in contrast to the usual method of preparation, i.e. incipient wetness, in which the cobalt oxide is dispersed as a second phase on the  $ZrO_2$ .

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